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**Applicant(s):** DOW GLOBAL TECHNOLOGIES INC.

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STRETCH FABRICS WITH IMPROVED CHEMICAL  
RESISTANCE AND DURABILITY  
BACKGROUND AND SUMMARY OF THE INVENTION

The present invention relates to stretch fabrics. In one aspect, the invention relates to stretch fabrics comprising synthetic and natural fibers while in another aspect, the invention relates to such fabrics comprising crosslinked, heat-resistant elastic fibers capable of withstanding dyeing and heat-setting processes. The crosslinked, heat-resistant elastic fibers are useful in various durable or repeated-use fabric applications such as, but not limited to, clothing, undergarments, sports apparel and upholstery. The crosslinked, heat-resistant elastic fibers can be conveniently formed into fabrics using well-known techniques such as, for example, by using co-knitting techniques with cotton, nylon, and/or polyester fibers.

A material is typically characterized as elastic if it has a high percent elastic recovery (that is, a low percent permanent set) after application of a biasing force. Ideally, elastic materials are characterized by a combination of three important properties, i.e., (i) a low percent permanent set, (ii) a low stress or load at strain, and (iii) a low percent stress or load relaxation. In other words, there should be (i) a low stress or load requirement to stretch the material, (ii) no or low relaxing of the stress or unloading once the material is stretched, and (iii) complete or high recovery to original dimensions after the stretching, biasing or straining is discontinued.

To be used in the durable fabrics, the fibers making up the fabric have to be, *inter alia*, stable during dyeing and heat setting processes. For an elastic polyolefin fiber to be stable under dyeing and heat-setting conditions, it must be crosslinked. These fibers can be crosslinked by one or more of a number of different methods, e.g., e-beam or UV irradiation, silane or azide treatment, peroxide, etc., some methods better than others for fibers of a particular composition. For example, polyolefin fibers that are irradiated under an inert atmosphere (as opposed to irradiated under air) tend to be highly stable during dyeing processes (that is, the fibers do not melt or fuse together). The addition of a mixture of hindered phenol and hindered amine stabilizers further stabilized such fibers at heat setting conditions (200-2100C).

Lycra®, a segmented polyurethane elastic material manufactured by E. I. du Pont de Nemours Company, is currently used in various durable stretch fabrics. Lycra, however, is not stable at the typical high heat-setting temperatures (200-210°C) used for polyethylene

terephthalate (PET) fiber . Moreover, and similar to ordinary uncrosslinked polyolefin-based elastic materials, Lycra fabrics tend to lose their integrity, shape and elastic properties when subjected to elevated service temperatures such as those encountered in washing, drying and ironing. As such, Lycra can not be easily used in co-knitting applications with high temperature fibers such as polyester fibers.

It has been discovered that elastic fabrics can be formed which are capable of surviving treatment that other elastic fabrics do not survive. Particularly the fabrics of the present invention can survive one or more of the following treatments: a) exposure to a 10% by weight sodium hypochlorite solution for a period of at least 90 minutes at a temperature of at least 140°F; b) exposure to a 5% by weight permanganate solution for a period of at least 90 minutes at a temperature of at least 140°F; c) 50 cycles of industrial laundering at temperatures of at least 65°C d) 20 cycles of drycleaning with perchloroethylene using AATCC test method 158; or e) mercerization under caustic conditions (28-33 Baumé or about 20% NaOH or more) at temperatures of at least about 60°C for 60 seconds or longer; wherein "surviving" means that the fabric after treatment exhibits growth of less than about 20%, preferably less than about 10%, and more preferably less than about 8%.

#### BRIEF DESCRIPTION OF THE FIGURES

The Figure 1 is a photograph of four heavy weight, denim fabric samples comprising fiber made from AFFINITY ethylene/1-octene copolymer. Each sample was subjected to a different stone wash protocol, i.e., the first (or top) sample to a vintage wash, the second to an antique wash, the third to a destructive wash, and the fourth (or bottom) sample to a bleach-out wash. The stretch properties of each sample after the washing protocol were essentially the same as the stretch properties before the washing protocol. The dark blue patch on top of the first or top sample is the color of each sample before it was stone washed.

Figure 2 is a Scanning Electron Microscopy (SEM) image of a Speedo swimsuit after a five-month wear test. The suit is of a tricot warp knit structure made with a chlorine-resistant Lycra<sup>™</sup> fiber.

Figure 3 is an SEM image of the swimsuit of Figure 2 showing the loop structure under enhanced magnification.

Figure 4 is a SEM image of a Speedo swimsuit after a four-month wear test. The suit is of a weft knit single jersey structure made with a crosslinked AFFINITY ethylene/1-octene copolymer fiber.

Figure 5 is an SEM image of the swimsuit of Figure 4 showing the loop structure under enhanced magnification.

#### DESCRIPTION OF THE PREFERRED EMBODIMENTS

One aspect this invention is an elastic article such as fabric or an assembled garment that comprises a heat-resistant, crosslinked elastic fiber and an inelastic fiber, which article is capable of surviving harsh treatment. In one embodiment, the article is a durable stretch fabric made and processed from one or more crosslinked, heat-resistant olefin elastic fibers. The fabrics can be made by any process, e.g., weaving, knitting, etc., and from any combination of elastic and inelastic ("hard") fibers. These fabrics exhibit excellent chemical, e.g., chlorine, resistance and durability, e.g., they retain their shape and feel ("hand") over repeated exposure to service conditions, e.g., washing, drying, etc. For example, in one embodiment the article (for example fabric or an assembled garment) has a change in elasticity not in excess of about 10% and/or retains no more than about 50% of its growth more preferably no more than about 20% of its growth, more preferably no more than about 10% of its growth and most preferably no more than about 8% of its growth after exposure to a 5% by weight permanganate solution for a period of at least 90 minutes at a temperature of at least 140°F. In another embodiment, the article retains at least about 10% of its elasticity and/or no more than 50% of its growth, more preferably no more than 20% of its growth, most preferably no more than about 10% of its growth after exposure to a 10% by weight hypochlorite solution for a period of at least 90 minutes at a temperature of at least 140°F. In yet another embodiment, the article survives a treatment of 50 cycles of industrial laundering at temperatures of at least 65°C. In still another embodiment the article survives 20 cycles of drycleaning with perchloroethylene using AATCC test method 158. In yet another embodiment, the article survives) mercerization under caustic conditions (28-33 Baumé or about 20% NaOH or more) at temperatures of at least about 60°C for 60 seconds or longer. Preferably the article can survive a combination of two or more of the treatments. It is also preferred that the article be able to survive stone washing in combination with one or more of these treatments.

The elastic fibers are preferably crosslinked, heat-resistant olefin elastic fibers. Such fibers include ethylene polymers, propylene polymers and fully hydrogenated styrene block copolymers (also known as catalytically modified polymers). The ethylene polymers include the homogeneously branched and the substantially linear homogeneously branched ethylene polymers as well as ethylene-styrene interpolymers. The other fibers of the fabric can vary widely, and they include virtually all known natural and synthetic fibers, particularly inelastic fibers. Typical of these other fibers are cotton, wool, silk, nylon, polyester, and the like. Usually the crosslinked, heat-resistant olefin elastic fibers comprise a minority of the fabric on a weight basis.

The fabrics of this invention include fabrics such as (i) a stone-washed elastic cotton fabric, (ii) a dye-stripped elastic nylon fabric, (iii) a brilliant-colored, dyed elastic polyester (e.g., PET) fabric, (iv) a dry-cleaned elastic fabric (e.g., a fabric that has been exposed to perchloroethylene), and (v) a chlorine- or bromine-exposed elastic fabric comprising one or more of polyester, nylon and cotton. All of these fabrics are known to require harsh and stringent processes that utilize chemicals and conditions that would degrade most conventional stretch fabrics because these chemicals and conditions would degrade the stretch fiber component of these fabrics. The fabrics of this invention, however, comprise a stretch fiber that is particularly resistant to such degradation and as such, the fabric containing these fibers exhibits surprising durability and chemical resistance.

"Fiber" means a material in which the length to diameter ratio is greater than about 10. Fiber is typically classified according to its diameter. Filament fiber is generally defined as having an individual fiber diameter greater than about 15 denier, usually greater than about 30 ~~denier~~. ~~Fine~~ denier fiber generally refers to a fiber having a diameter less than about 15 denier. ~~Microdenier~~ fiber is generally defined as fiber having a diameter less than about 100 microns ~~denier~~.

"Filament fiber" or "monofilament fiber" means a single, continuous strand of material of ~~indefinite~~ (i.e., not predetermined) length, as opposed to a "staple fiber" which is a discontinuous ~~strand~~ of material of definite length (i.e., a strand which has been cut or otherwise divided into ~~segments~~ of a predetermined length).

The term "heat resistant" as used herein refers to the ability of an elastic polymer or elastic polymer composition in the form of fiber to pass the high temperature heat setting and dyeing tests described herein.

The term "survive" when used in reference to the fabric or article "surviving" a particular treatment means that the fabric exhibits growth of less than 20%, preferably less than about 10% and more preferably less than about 8% in both the warp and weft direction after the particular treatment has been conducted.

The term "growth" means residual elongation, or the amount the fabric lengthens after applying a load over a given length of time and allowing recovery, expressed as a percentage of the initial fabric dimension. Growth can be determined using ASTM D3107.

The term "treatment" means one or more of a) exposure to a 10% by weight sodium hypochlorite solution for a period of at least 90 minutes at a temperature of at least 140°F; b) exposure to a 5% by weight permanganate solution for a period of at least 90 minutes at a temperature of at least 140°F; c) 50 cycles of industrial laundering at temperatures of at least 65°C d) 20 cycles of drycleaning with perchloroethylene using AATCC test method 158; or e) mercerization under caustic conditions (28-33 Baumé or about 20% NaOH or more) at temperatures of at least about 60°C for 60 seconds or longer.

The term "elastic article" is used in reference to shaped items, while the term "elastic material" is a general reference to polymer, polymer blends, polymer compositions, articles, parts or items.

"Elastic" means that a fiber will recover at least about 50 percent of its stretched length after the first pull and after the fourth to 100% strain (doubled the length). Elasticity can also be described by the "permanent set" of the fiber. Permanent set is the converse of elasticity. A fiber is stretched to a certain point and subsequently released to the original position before stretch, and then stretched again. The point at which the fiber begins to pull a load is designated as the percent permanent set. "Elastic materials" are also referred to in the art as "elastomers" and "elastomeric". Elastic material (sometimes referred to as an elastic article) includes the polyolefin polymer itself as well as, but not limited to, the polyolefin polymer in the form of a fiber, film, strip, tape, ribbon, sheet, coating, molding and the like. The preferred elastic material is fiber. The elastic material can be either cured or uncured, radiated or unirradiated, and/or crosslinked or uncrosslinked. For heat reversibility, the elastic fiber must be substantially

crosslinked or cured. For purposes of this invention, an elastic article is one that comprises elastic fiber.

"Nonelastic material" means a material, e.g., a fiber, that is not elastic as defined above.

Suitable fibers for use in the present invention are disclosed in US 6,437,014. As described in that reference, the fibers can be formed by many processes known in the art, for example the fibers can be meltblown or spunbond. Similarly, as taught in US 6,437,014, the fibers can be made from many different materials, including ethylene-alpha olefin interpolymers, substantially hydrogenated block polymers, styrene butadiene styrene block polymers, styrene-ethylene/butene-styrene block polymers, ethylene styrene interpolymers, polypropylenes, polyamides, polyurethanes and combinations thereof. The crosslinked homogeneously branched ethylene polymers described in that reference, particularly the substantially linear ethylene polymers, are particularly well suited for use in making articles of this invention.

These fibers may be used neat or may be combined into a yarn with an inelastic fiber such as cotton, wool, or synthetic material such as polyester or nylon. These fibers, whether neat or used with other material in a yarn, may be used alone or together with other yarns to make the fabric of the present invention. The fabric of the present invention can be made according to known fabrication methods such as weaving or knitting. A preferred fabric of the present invention is denim, as many current processes for treating denim are too harsh for current elastic fabrics. Thus, denim made from elastic fabric of the present invention will be able to survive the current treatments.

The fabric of the present invention can then be used to make garments. Examples of garments which can be advantageously made from the fabric of the present invention include swimwear and uniforms, particularly rental uniforms which are subject to industrial laundering.

The following examples are to illustrate the invention, and not to limit it. Ratios, parts and percentages are by weight unless otherwise stated.

### EXPERIMENTAL

#### Fiber Descriptions:

Fiber made from Dow AFFINITY ethylene-octene copolymer (MI 3 g/10min, density 0.875 g/cc) 140 Denier crosslinked by e-beam (19.2 mrad)

Generic spandex

**Fabric Description:**

3 x 1 RHT (right-hand twill); 100% cotton warp, 94% cotton/6% Crosslinked AFFINITY filling.

**Example 1: Stone Washing**

The stones were white pumas ranging approximately between 2-4 inches in diameter.

The stones were soaked in the chemical solution for two (2) hours prior to testing.

**Stone Wash/Decolorize – Hypochlorite Formula**

<b>Process</b>	<b>Liquor Ratio</b>	<b>Water Temp (F)</b>	<b>Time (Min)</b>	<b>Chemical Quantity</b>	<b>Chemical</b>	<b>Comment</b>
Stonewash/ Hypochlorite	10:1	140	90	10% soln. 5.25% available Cl (stone soak)	Sodium Hypochlorite	3:1 Stone to Fabric ratio
Drain/Rinse	10:1	170	10			Rinse
Neutralize	10:1	170	20	0.5 g/l	Sodium Disulfite	
Drain/rinse						Rinse Hot Rinse Cold
Dry						Tumble Dry Low



Stone Wash/Decolorize – Permanganate Formula

Process	Liquor Ratio	Water Temp (F)	Time (Min)	Chemical Quantity	Chemical	Comment
Stonewash/ Potassium Permanganate	10:1	140	90	5% soln. (stone soak)	Potassium Permanganate	3:1 Stone to Fabric ratio
Drain/Rinse	10:1	170	10			Rinse
Neutralize	10:1	170	20	0.5 g/l	Sodium Bisulfite	
Drain/rinse						Rinse Hot Rinse Cold
Dry						Tumble Dry Low

**Test Results:**

To understand the effects of stone washing on spandex, a sample of stretch denim comprising spandex was run in parallel with a sample of stretch denim comprising AFFINITY fiber. Although the properties of the two fabrics cannot be compared directly (the fabrics are of slightly different constructions), the data does show, however, property degradation in spandex-based denims and property retention in AFFINITY-based denims.

Test Procedures	AFFINITY Denim		Spandex Denim	
	Length	Width	Length	Width
Fabric Dimensional Change (AATCC 135) After Stone Wash, Chlorine Bleach	-2.2%	-1.6%	4.9%	-10.2%
Fabric Dimensional Change (AATCC 135) After Stone Wash, Permanganate	-2.6%	-1.7%	-5.1%	-10.5%
<b>Stretch and Recovery Comparison (ASTM D6614)</b>	<b>Stretch</b>	<b>Growth</b>	<b>Stretch</b>	<b>Growth</b>
As Received	7.0%	2.9%	17.3%	4.5%
After 1x Stone Wash, Chlorine Bleach	7.3%	3.5%	28.3%	8.0%
After 1x Stone Wash, Permanganate	7.5%	3.5%	29.9%	10.1%

Denim fabric containing AFFINITY fiber did not have any significant change in stretch properties. When a commercially available spandex containing stretch fabric was subjected to the hypochlorite and permanganate washes, it exhibited deterioration in stretch properties and dimensional stability.

#### Example 2: Stripping Agents

Chemical Reduction by 1 g/L Sodium Hydrosulfite (Dye Stripping), 100°C/212°F, 1 hour:

Dye Stripping is a process to chemically remove color from fabric for redyeing. This test was performed as sodium hydrosulfite is a commonly used dye stripping agent. Since published research has shown some sensitivity on the part of elastomeric fibers to dye-stripping. Dyers prefer to work with a fiber that can withstand a stripping bath rather than one that will not.

#### Fiber Description:

Fiber made from Dow AFFINITY EG 8200 (MI 5 g/10min, density 0.870 g/cc) 70 Denier crosslinked by e-beam (32 mrad)

Dupont Lycra 70 Denier

Dupont Lycra – Chlorine Resistant 70 Denier

Fiber Test Data

	<b>AFFINITY</b>	<b>Lycra</b>	<b>Lycra-CR</b>
Ultimate Elongation After Treatment (%)	276.68	334.94	297.26
% Difference against as received	-16%	-23%	-28%
Breaking Load After Treatment (g)	32.35	49.21	47.37
% Difference against as received	-53	-43	-33

Example 3: Swimming Pool Water

100 ppm Sodium Hypochlorite (Chlorine Bleach), 50°C/120°F, 24 hours:

This accelerated test was performed as the hypochlorite ion is responsible both for bleaching and fiber damage in textiles, and it is also a chief cause in the degradation of fibers by swimming pool water. This level of chlorine was found by ruggedness testing to be roughly equivalent to the amount of exposure that would cause failure in a chlorine resistant Lycra® swimsuit after five months of use in which the suit was worn three times per week.

Fiber description:

Fiber made from Dow AFFINITY EG 8200 (MI 5 g/10min, density 0.870 g/cc) 70 Denier crosslinked by e-beam (32 mrad)

Dupont Lycra 70 Denier

Dupont Lycra – Chlorine Resistant 70 Denier

	<b>AFFINITY</b>	<b>Lycra</b>	<b>Lycra-CR</b>
Ultimate Elongation After Treatment (%)	250.23	125.83	206.50
% Difference against as received	-24%	-71%	-50%
Breaking Load After Treatment (g)	38.46	2.12	15.19
% Difference against as received	-44%	-98%	-79%

Example 4: Wear Test

Fiber description:

Fiber made from Dow AFFINITY EG 8200 (MI 5 g/10min, density 0.870 g/cc) 70 Denier crosslinked by e-beam (32 mrad)

A Speedo suit made of a two bar tricot construction with nylon and conventional Lycra spandex was obtained that displayed almost complete disintegration of the spandex component. Additionally new Speedo suits containing chlorine resistant Lycra spandex were purchased, and a swimsuit was constructed using weft knit polyester (about 88% by weight)/Dow AFFINITY fiber (about 12% by weight) fabric.

After a five-month wear trial test, the chlorine resistant suit displayed localized degradation. Scanning Electron Microscopy (SEM) images (Figures 2 and 3) revealed that this degradation involved only the spandex filaments which were heavily degraded while the nylon filaments were untouched.

In contrast to the chlorine resistant spandex, the crosslinked AFFINITY elastomeric yarn contained in a similar swimsuit used in a four month wear trial displayed no degradation (Figures 4 and 5). No significant bagging of the AFFINITY suit was found present and the suit was found to be functional in all ways with exception of the polyester yarn's propensity to stain readily when exposed to zinc oxide sun block, sun tan lotion and oil.

After completion of the wear trial, the AFFINITY suit was washed using the machine wash/warm tumble dry low cycle. The suit improved in appearance due to removal of stains and dirt accumulated over the period of the wear trial. After washing, the suit continued to fit well without bagging or excess shrinkage.

#### Example 5: Laundering

Stretch Properties of Fabric Containing AFFINITY Crosslinked Fibers:

Fabric description: 3 X 1 LHT (left-hand twill); 100% Nylon T-66 warp, 84% cotton/16% Dow AFFINITY EG 8200 (MI 5 g/10min, density 0.870 g/cc) 70 Denier crosslinked by e-beam (22.4 mrad) filling.

Laundry Method	Conditions	Fabric Stretch, % weft direction (ASTM-D-6614-00)		
		1 cycle	25 cycles	50 cycles
MWH TDH SIM	From AATCC Test Method 135 <ul style="list-style-type: none"> <li>• machine wash hot (normal cycle, 12 minutes), 140°F</li> <li>• tumble dry high, 160°F</li> <li>• steam iron medium, 300°F</li> </ul>	66.6	70.2	73.0
MWH TDH SIM With Chlorine (CLOROX®)	From AATCC Test Method 135 <ul style="list-style-type: none"> <li>• machine wash hot (normal cycle, 12 minutes), 140°F</li> <li>• tumble dry high, 160°F</li> <li>• steam iron medium, 300°F</li> </ul>	65.0	70.1	74.6
MWH TDH SIM With Non-Chlorine Bleach (CLOROX 2®)	From AATCC Test Method 135 <ul style="list-style-type: none"> <li>• machine wash hot (normal cycle, 12 minutes), 140°F</li> <li>• tumble dry high, 160°F</li> <li>• steam iron medium, 300°F</li> </ul>	64.1	66.4	71.0

The data in the above table demonstrates that the fabric experiences minimal change over 1 to 50 cycles.

Although the invention has been described in considerable detail through the preceding embodiments, this detail is for the purpose of illustration. Many variations and modifications can be made on this invention without departing from the spirit and scope of the invention as described in the following claims. All U.S. patents and allowed U.S. patent applications cited above are incorporated herein by reference.

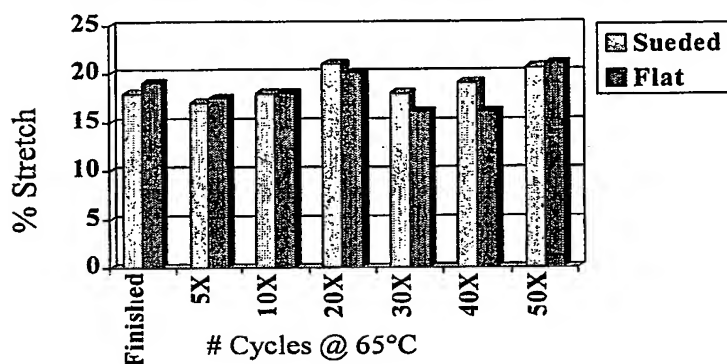
Example 6: Industrial Laundering:

Fabric Description: 2X1 right hand twill; 65%/35% Polyester/Cotton Warp; 96% Cotton/4% XLA Filling Yarn

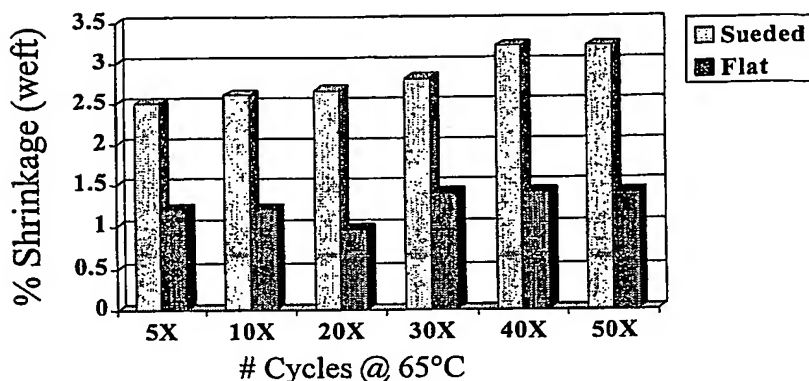
Fabric was prepared, dyed and finished using in a continuous, thermosol dyeing range. The maximum temperature of during processing was 440°F. Finished fabric was cut and sewn into pant legs and subjected to industrial laundering at 65°C. Fabric stretch and dimensional stability were measured after 5, 10, 20, 30, 40, and 50 cycles. Fabric stretch and dimensional stability showed no significant change after multiple cycles in industrial laundering.

## Laundering data

### Thermosol Dyeing of DOW XLA<sup>®</sup> Based Fabrics



## Laundering Data Thermosol Dyeing of DOW XLA<sup>®</sup> Based Fabrics



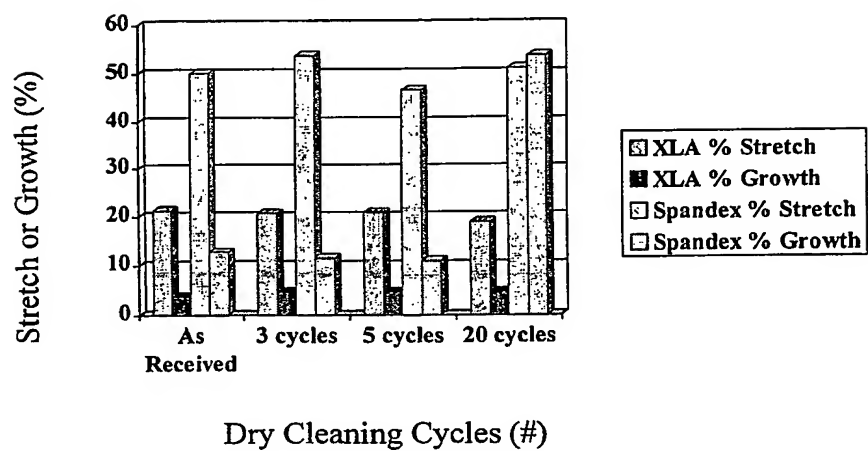
### Example 7: Drycleaning

Fabric Description: Plain Weave Chambray Fabric; 100% Cotton Warp; 94%/6% Cotton/XLA Filling

Fabric was prepared and finished on a continuous range. The maximum temperature during processing was 365°F. Finished fabric was cut and sewn into tubes. In addition, Chambray shirts containing spandex were purchased for comparison. Shirts and tubes were subjected to multiple drycleaning cycles using perchloroethylene as the solvent. Stretch and growth were measured after 3, 5, and 20 cycles. Fabric stretch and growth for the XLA fabric were not significantly changed after multiple drycleaning cycles. However, the spandex fabric experienced excessive growth after 20 drycleaning cycles.



## Effects of Dry Cleaning on % Stretch and % Growth



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